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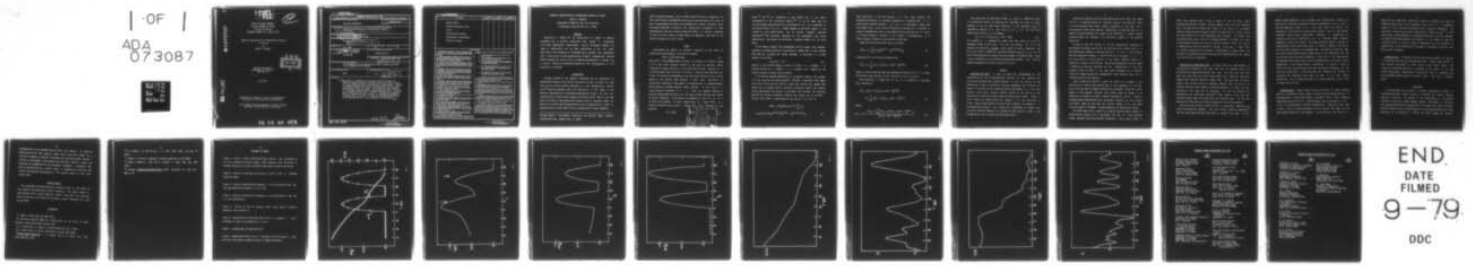

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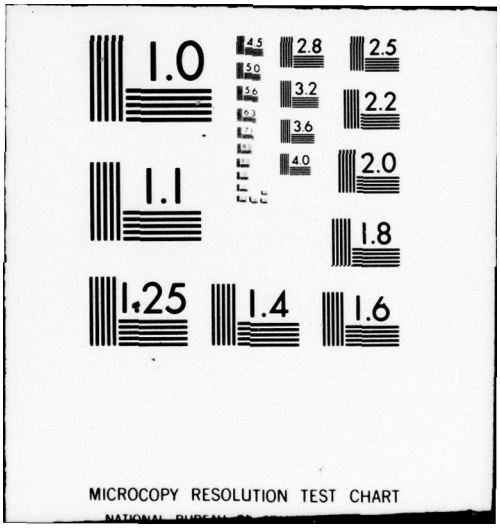
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Numerical Deconvolution of Photoelectron Spectra of Liquids

by
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NUMERICAL DECONVOLUTION OF PHOTOELECTRON SPECTRA OF LIQUIDS

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Abstract

Described is a method for the deconvolution or removal of spectral broadening due to electron interactions with solvent in liquid-phase ultraviolet photoelectron spectroscopy. Certain differences between this usage of deconvolution and its usual applications in pes are noted. Application of the technique is illustrated using synthetic and experimental data. Possible uses include recovering the "original" photoemission spectrum before broadening by liquid as well as studying the mechanism of energy loss in solvent. Effects of unscattered electrons on the interpretation of this procedure are discussed.

INTRODUCTION

Although a theory[1] of the spectral broadening due to interactions of photoemitted electrons with liquid in bulk phase has been available for some time, and its validity recently confirmed on a more mathematically rigorous basis,[2] there has remained a gap in the application of this theory to the detailed interpretation of liquid-phase photoelectron spectra. Up until now, interpretation of these electron energy distribution curves (EDC's) has been made on the basis of semi-quantitative measures such as threshold energy for emission, or by more sophisticated measures such as superposition of EDC's or second derivative curves (SDC's) which, although able to reveal something

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about the detailed spectra, rely on certain simplifications or assumptions for their justification (although SDC curves may be corrected empirically, band by band, to approximate the results of the deconvolution method described here). The present technique is related to the deconvolution procedures[3] of other spectroscopies, and can be used to remove the effects of spectral broadening in liquid, at least insofar as these effects are adequately described by the theory of energy loss in bulk liquid.

THEORY

Liquid-phase pes spectra of electrons originating in the "bulk" of solution can be described by a convolution integral,

$$f^m(T) = \int_{-\infty}^{\infty} f^t(x)B(T,x)dx \qquad (1)$$

where $f^m(T)$ is the measured partial current of electrons of kinetic energy T, f^t is the "true" electron current at the point of emission, before scattering by liquid, and $B(T,x)$ is the convolution function. Equation (1) is a slightly generalized form of Eq. (1) of Ref. 3, which has been modified here to take into account the dependence of the convolution function on the kinetic energy of the electron upon emission. A more usual application of deconvolution techniques is the removal of broadening due to nonmonochromatic photon sources or nonideal electron energy analysis. In this case, B is usually given as a function of $(T - x)$. There are numerous methods of obtaining approximate solutions to Eq. (1), many of them involving some degree of implicit smoothing. It was found to be expedient to solve Eq. (1) by a so-called direct method, in which deconvolution of a given $f^m(T)$, as a digitally tabulated array, yields a unique $f^t(T)$ array, according to the equation,

$$\bar{f}^t = B^{-1}\bar{f}^m \qquad (2)$$

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where $\bar{f}^t$ and $\bar{f}^m$ are interpreted as data vectors and B the matrix corresponding to the convolution function.[3,4] It can be shown that the recursive method described below for the solution of Eq. (1) corresponds to the solution given in Eq. (2). A major drawback to the direct method is the problem of noise amplification. Use of Fourier Transform smoothing techniques[5,6] were absolutely necessary for the experimental data before deconvolution. More details of the Fourier Transform method will be given below.

In the theory of Nemec,[1] the instantaneous loss of energy from energetic electrons to solvent molecules in the course of random walk to the surface where they are collected and energy analyzed, is described in a single equation of the form,

$$d(T_\ell)/dt = - \alpha v^n \tag{3}$$

where T_ℓ is the electron energy in liquid, t is time, α is a constant, v is electron velocity ($T_\ell = 2^{-1} m_e v^2$), n is a constant which depends on the physical process of energy loss to solvent.

If an electron becomes thermalized ($T_\ell \rightarrow 0$) before reaching the surface, it is not counted by the energy analyzer. Electrons originating from deeper within the solution are less likely to reach the surface and undergo more energy loss, on average, than do electrons originating nearer the surface. Taking these factors into account in the random walk problem, the following equation for the EDC of electrons reaching the surface can be derived.[1] From Eq. (16) of Ref. 1, generalized for any value of $n \neq 3$, this is:

$$dN/dT_s = (QCF_a/K^2)(2\lambda/3\pi)^{1/2} \int_{T_s}^{\infty} D(T_g)$$

$$\cdot\{(m_e/(3-n)\alpha)(2/m_e)^{(3-n)/2}(T_g^{(3-n)/2} - T_s^{(3-n)/2})\}^{1/2} dT_g \tag{4}$$

where $dN(T_s)/dT_s$ is the EDC function, Q is the cross section for photoelectron emission, C is emitter concentration, F is photon flux, K is a normalization constant (see Ref. 1, Eq. (5)), λ is mean-square random walk step length, $D(T_g)$ is the initial distribution of electrons as a function of energy of photoemission (this is the unknown function which we wish to find), T_g is kinetic energy of electron at the instant of photoemission, T_s is kinetic energy of electron at the surface of the liquid.

Combining the constant terms into K_1, we obtain

$$dN/dT_s = K_1 \int_{T_s}^{\infty} D(T_g)(T_g^{(3-n)/2} - T_s^{(3-n)/2})^{1/2} dT_g \tag{5}$$

Transposing this into finite difference form:

$$E(T_s) = K_1 \sum_{j=0}^{N} D(T_s + j\Delta T)((T_s + j\Delta T)^p - T_s^p)^{1/2}\Delta T \tag{6}$$

where E is the discrete EDC data set tabulated at intervals of ΔT, N is taken sufficiently large that $D(T_s + j\Delta T) = 0$ for all $j \geq N$, p is $(3-n)/2$.

Given that $E(T_s)$ is known for all T_s, it is possible to calculate $D(T_g)$ recursively:

$$E(T_s) = D(T_s + \Delta T)\, K_1((T_s + \Delta T)^p - T_s^p)^{1/2}\Delta T + K_1 \sum_{j=2}^{N} D(j\Delta T + T_s)((T_s + j\Delta T)^p - T_s^p)^{1/2}\Delta T \tag{7}$$

whence,

$$D(T_s + \Delta T) = \frac{E(T_s) - K_1 \sum_{j=2}^{N} D(T_s + j\Delta T)((T_s + j\Delta T)^p - T_s^p)^{1/2}\ \Delta T}{K_1((T_s + \Delta T)^p - T_s^p)^{1/2}\Delta T} \tag{8}$$

Thus, given that for sufficiently large T_s, $D(T_g)$ is identically zero, it is possible to use Eq. (8) recursively to calculate the array of $D(T_g)$. Although it has not been shown explicitly in the equations, $D(T_g)$ is tabulated at intervals of ΔT, from large T_g to $T_g = \Delta T$. For the first nonzero value of $E(T_s)$, Eq. (8) becomes:

$$D(T_s + \Delta T) = E(T_s)/K_1((T_s + \Delta T)^p - T_s^p)\Delta T \qquad (9)$$

The constant K_1 is automatically taken into this value of D and all subsequent values in the D array. This has two effects: (1) it is not necessary to calculate K_1 a priori, which would be nearly impossible, and (2) K_1 will differ in numerical value when different values of p (or n) are used in the deconvolution, making the results appear to different scale (there is also a difference of scale between deconvolution and SDC). This recursive method corresponds to the exact solution of Eq. (2).

RESULTS

Simulated EDC data. In order to check the correspondence of the deconvolution, Eq. (8), to the generating function, Eq. (6), synthetic EDC curves were generated from a known D function. To test the ability of the deconvolution to work on functions of arbitrary complexity, the D function was chosen to be portions of two inverted parabolas superimposed on a flat zero baseline. Use of such a function enables testing of the ability of the deconvolution to separate closely spaced and rather narrow bands. Figure 1, curve A shows the D function which was assumed in all the simulations to follow. Curve B shows the EDC calculated from A using Eq. (6) assuming $p = 1$ (corresponding to $dT_1/dt = -\alpha v$). Without the introduction of random noise or Fourier Transform smoothing, and assuming the same value of p used in the EDC generation, the D function was reproduced exactly.

Contrast this behavior with the (uncorrected) SDC of the same data shown in Fig. 2. As shown previously for simulated Gaussian distribution,[7] there is a shifting toward lower kinetic energy, broadening of the peak, and a rising baseline toward lower energies. Nevertheless, the SDC does seem to be capable of good performance with respect to the difference in energy between successive peaks, and reproduces the qualitative aspects of the spectrum rather well.

In order to test the ability of the EDC deconvolution technique to discriminate the energy loss law, Eq. (3), EDC data with a certain value of p were deconvoluted assuming a different value of p. In Fig. 3, the original data were generated assuming p = 1 and fit with Eq. (8) assuming p = 2. In this case, there is a rising portion at low kinetic energies similar to, though not as severe as the SDC case. The relative magnitude of the two peaks is affected, but the peak positions are not. Deconvolution of real data assuming a value of p which is too large would therefore be a difficult situation to detect because the systematically high baseline could not be differentiated from real emission.

Figure 4 shows the situation in which the value of p assumed in the deconvolution is smaller than actually used in the generation of the EDC. Here we see that areas of negative emission are calculated. Since this cannot happen in a real physical situation, this effect might be thought to be diagnostic of a choice of p which is smaller than physical reality. Actually, disproportionate contributions from unscattered electrons can also produce this effect. Again we see that the relative heights of the two peaks (initially equal) is affected. The peak energies are reassuringly accurate.

The results seen in Fig. 4 point to a crude but possibly usable method of determining the value of p (i.e., the energy loss law) in a real physical system. Necessary would be an emitter, preferably a pure liquid, which is

known from gas-phase data to have a region of very low (near zero) photoelectron emission at higher binding energies than the first emission band. One would have to choose such a liquid on the basis of a gas-phase spectrum showing an emission gap at least 2-3 eV wide (and, of course, having the other properties of a good liquid-phase pes prospect). By taking very accurate EDC data using a source of low-energy photons to minimize the yield of unscattered electrons, the value of p could be found at which the results of deconvolution yield zero emission in the gap. In any case, a lower limit on p might be obtained by this procedure. Unfortunately, as shown below, the existence of unscattered electrons may make such a determination of the energy loss law difficult or impossible.

Application to Experimental Data. In order to test the applicability of the deconvolution method to real data, EDC data for ethylene glycol and N-methylaniline reported previously[8] were used. The data were read from paper tape, where they had initially been stored, into a PDP-11/34 laboratory computer where they were stored on floppy disk. The data were originally taken as total current curves at a retarding potential grid and had to be differentiated once (with respect to retarding potential) to yield the EDC. Smoothing at this stage was not necessary. The zero of potential was determined by the capacitance technique.[8] These capacity curves had been recorded on paper tape at the same time as the emission data. All EDC and deconvolution data shown are already corrected for this offset potential.

Upon going from the EDC to either the SDC or deconvolute, smoothing was found to be absolutely necessary. The Fast Fourier Transform technique[5,6] with the sigma smoothing factors described by Lanczos[9] was used. In this

method, the EDC spectrum is first extended with baseline-level currents to obtain cyclic continuity required for Fourier transformation and to yield a data set numbering a power of 2, in the cases below, 512 points. The transform of a real array of 512 data points yields an array of 256 complex "frequency" space Fourier coefficients. Smoothing is accomplished by truncating this array of coefficients so as to eliminate higher frequency components in which noise resides. The sigma coefficient method is one way of accomplishing a "soft" cutoff which tends to minimize unwanted oscillations near the cutoff frequency in the smoothed data. The choice of cutoff was made by visually observing the tradeoff between noise in the deconvolute and the loss of detail due to excessive smoothing. In the case of the deconvolution of N-methylaniline, below, special care was needed because of the many closely spaced bands. It was always possible, nevertheless, to choose a range of cutoffs for which the peak energies were well defined and unchanging, and the noise was not excessive. In the cases below, the smoothing was effected by truncating the components above about the 40th of the 256 complex coefficients. This is roughly equivalent to a moving smoothing function with a window 0.2-0.5 eV wide.

Ethylene Glycol. Figure 5 shows a portion of the EDC of liquid ethylene glycol as a function of kinetic energy, corrected for the surface potential as described above. The data were taken as described[8] with a He(I) resonance lamp photon source of 21.2 eV. "Binding energy" can be calculated as (21.2 - T) eV. For ethylene glycol, the EDC is monotonically increasing (from right to left). Thus, it should be possible to find a value of the power law for which negative emission is not observed. It turns out that the choice of a

power loss law coefficient sufficiently large to eliminate the areas of "negative" emission seen in the deconvolute, Fig. 6 for p = 1, is impossibly large, p = 5 or 6, with severe distortion of the resulting deconvolute. The conclusion, reinforcing the earlier observations of Nemec et al.,[8] is that there exists a strong component of unscattered electrons in the EDC spectrum. The deconvolution method appears to be superior to the method of empirical correction of SDC's in that it is able to demonstrate (by the existence of negative areas in the deconvolute) the presence of unscattered electrons even when the EDC is monotonic.

N-Methylaniline. This material exhibits more obvious effects of emission of unscattered electrons than does ethylene glycol. Figure 7 shows the EDC of data recorded previously[8] at -44°C, using a He(I) photon source. The presence of unscattered electrons is indicated by the maxima in the high kinetic energy peaks. Deconvolution of such an EDC (Fig. 8) indicates even more pronounced negative peaks in the result than in the case of ethylene glycol.

CONCLUSION

It has been shown to be possible to perform deconvolution directly on liquid-phase EDC data without relying on corrected SDC analysis assuming Gaussian D function. The problem of the overabundance of unscattered electrons is seen to be a major drawback, however. The attempt at correction for unscattered electrons by superposition of corrected SDC derived Gaussian emission from bulk with a variable band due to unscattered electrons was shown previously to be effective in fitting the data,[8] though the physical

meaningfulness of this procedure may be called into question. An empirical modification of the bulk emission theory which gives more weight (as a function of energy) to electrons originating near the surface might provide a solution to the problem, though without much additional physical insight and requiring the introduction of extra adjustable parameters. Ultimately, what would be desired would be a unified theory of photoelectron emission from liquids, with detailed consideration of the discrete nature of the liquid surface layer.

ACKNOWLEDGMENTS

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CAPTIONS TO FIGURES

Figure 1. Curve A - Initial distribution $D(T_g)$; Curve B - EDC calculated by Eq. (6), as function of kinetic energy. Both functions were calculated as discrete functions, the actual tabulated values shown as dots on the curves.

Figure 2. Results of taking SDC (derivative) of Curve B in Fig. 1. Discrete points not shown.

Figure 3. Results of deconvolution assuming p = 1 for calculation of EDC (Eq. (6)) and deconvoluted assuming p = 2 (Eq. (8)).

Figure 4. Results of deconvolution assuming p = 2 for calculation of EDC and p = 1 for deconvolution.

Figure 5. Portion of EDC for ethylene glycol after Fourier Transform smoothing. Data from Ref. 8.

Figure 6. Deconvolution of glycol EDC data of Fig. 5. p assumed = 1. Little difference in results was observed for p = 0 or 2.

Figure 7. Smoothed EDC of N-methylaniline.[8]

Figure 8. Deconvolute of EDC in Fig. 7. Variation of p from value of 1 used here had little effect on extensive areas of "negative emission."

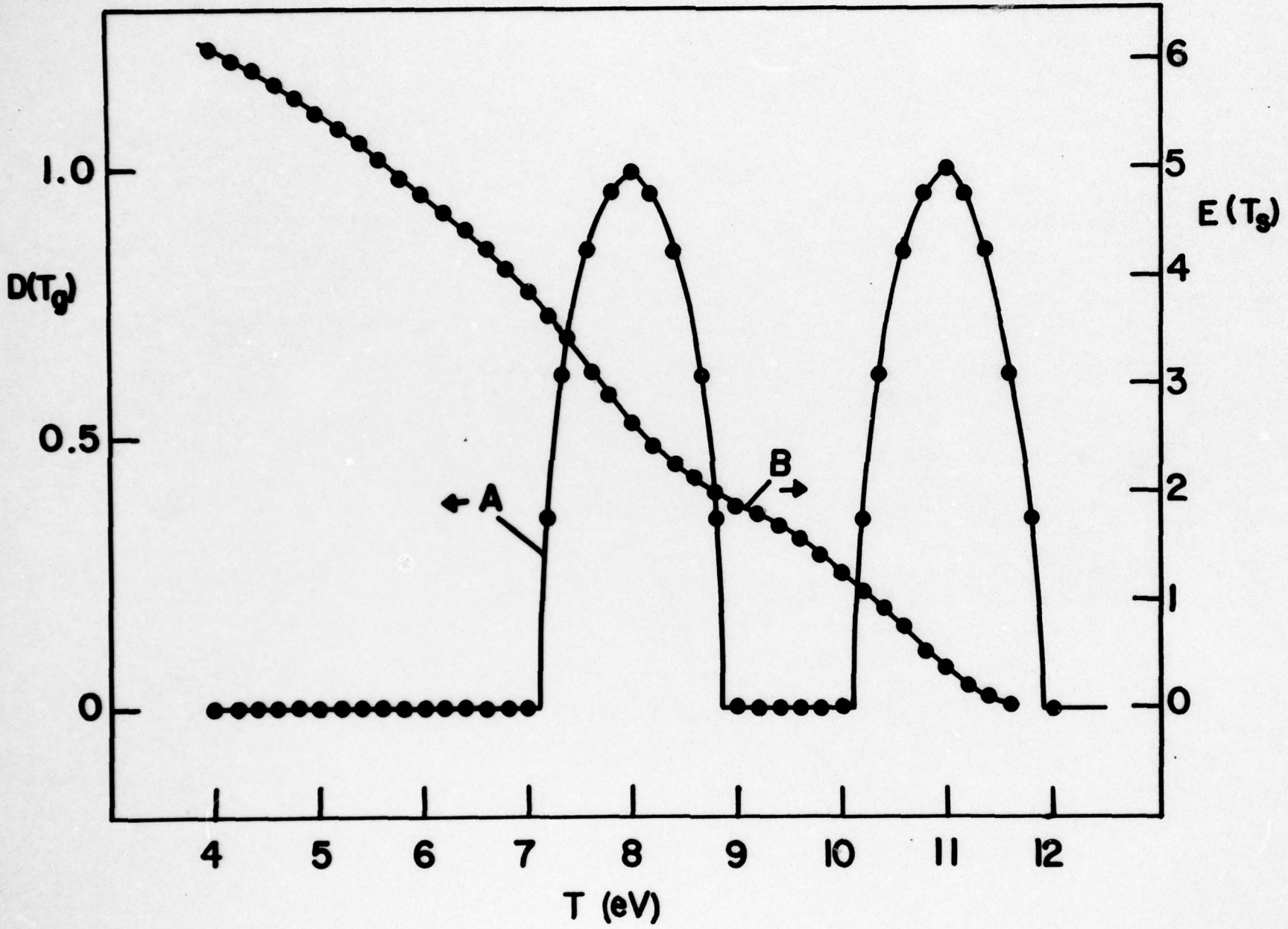
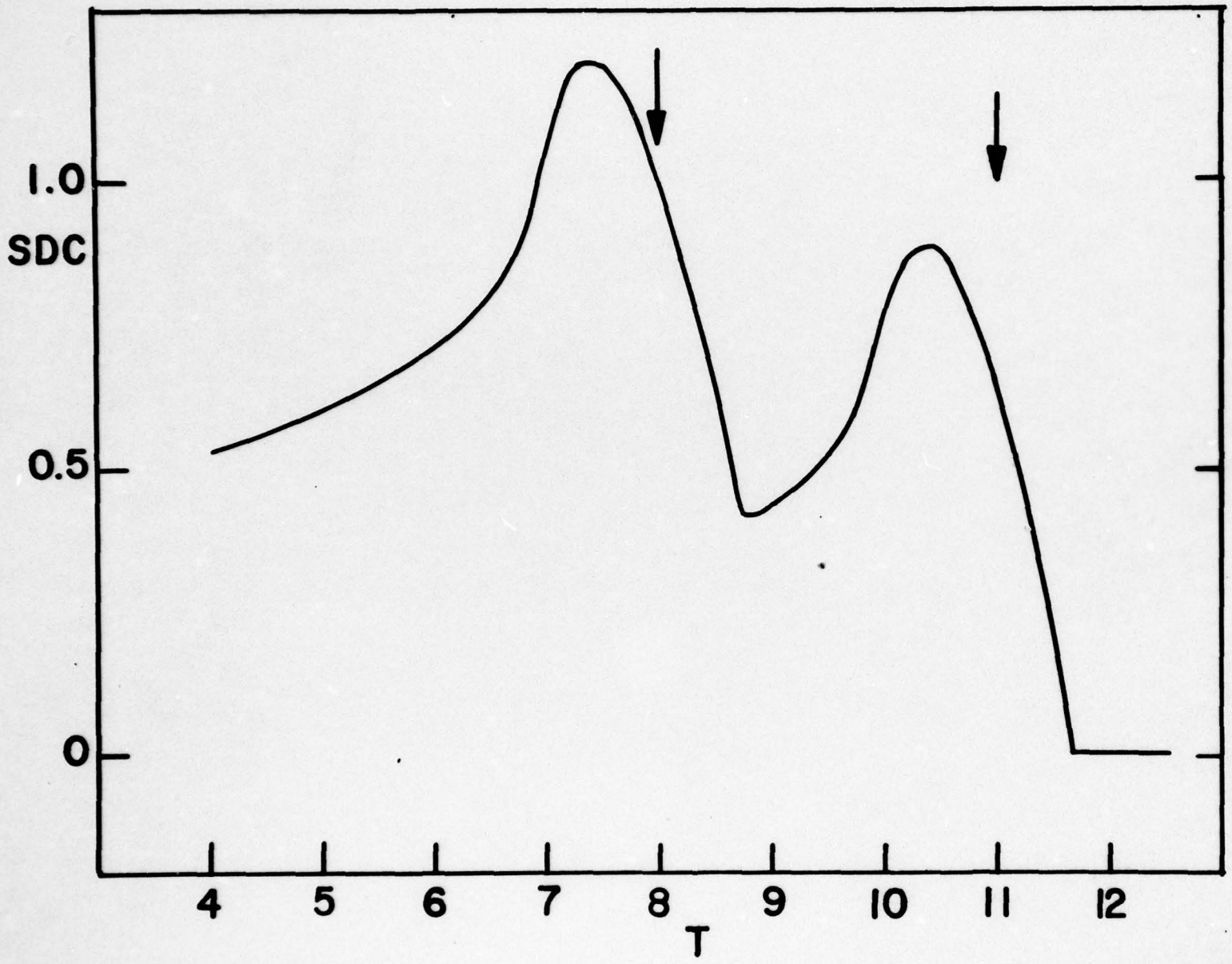


FIG. 1



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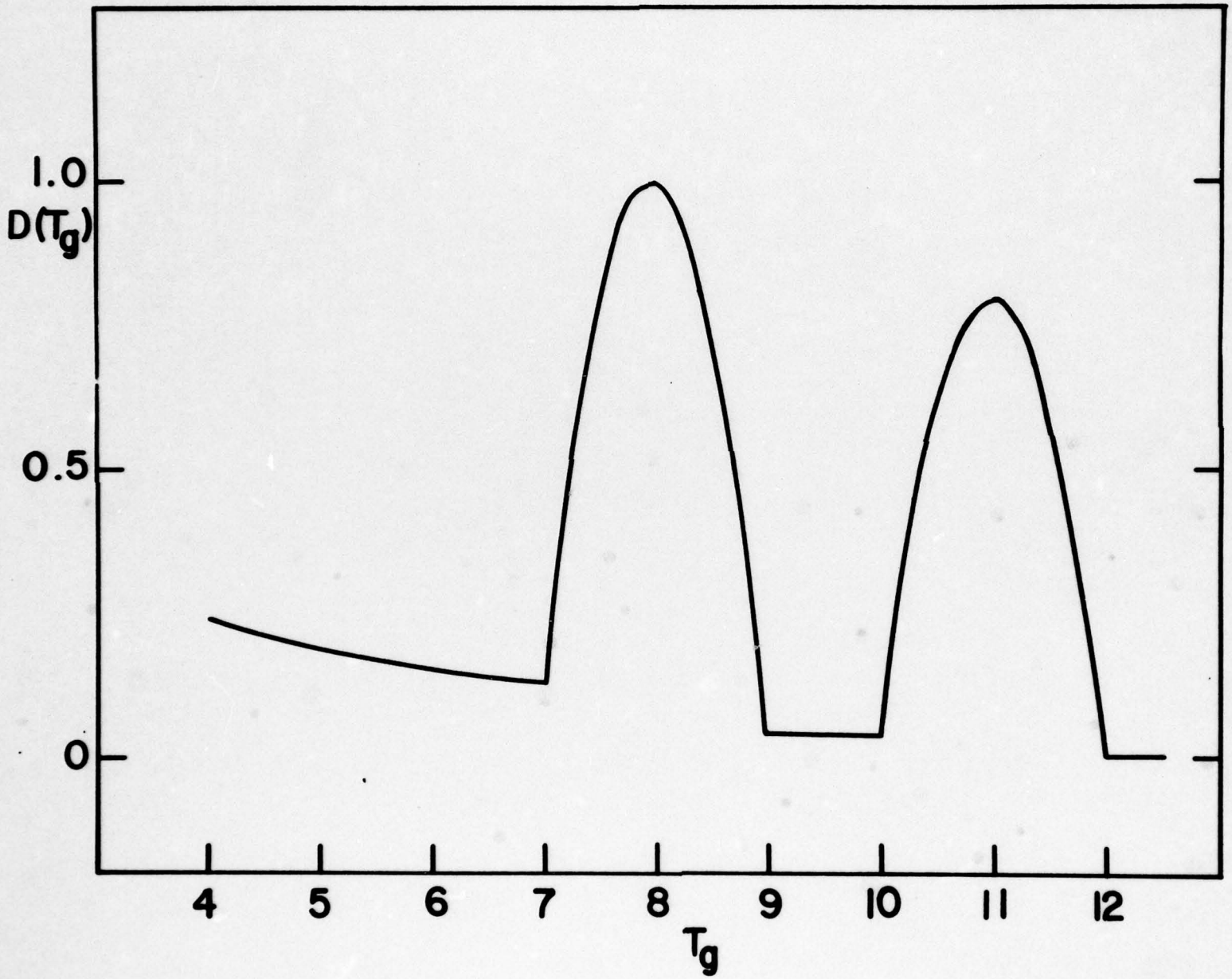

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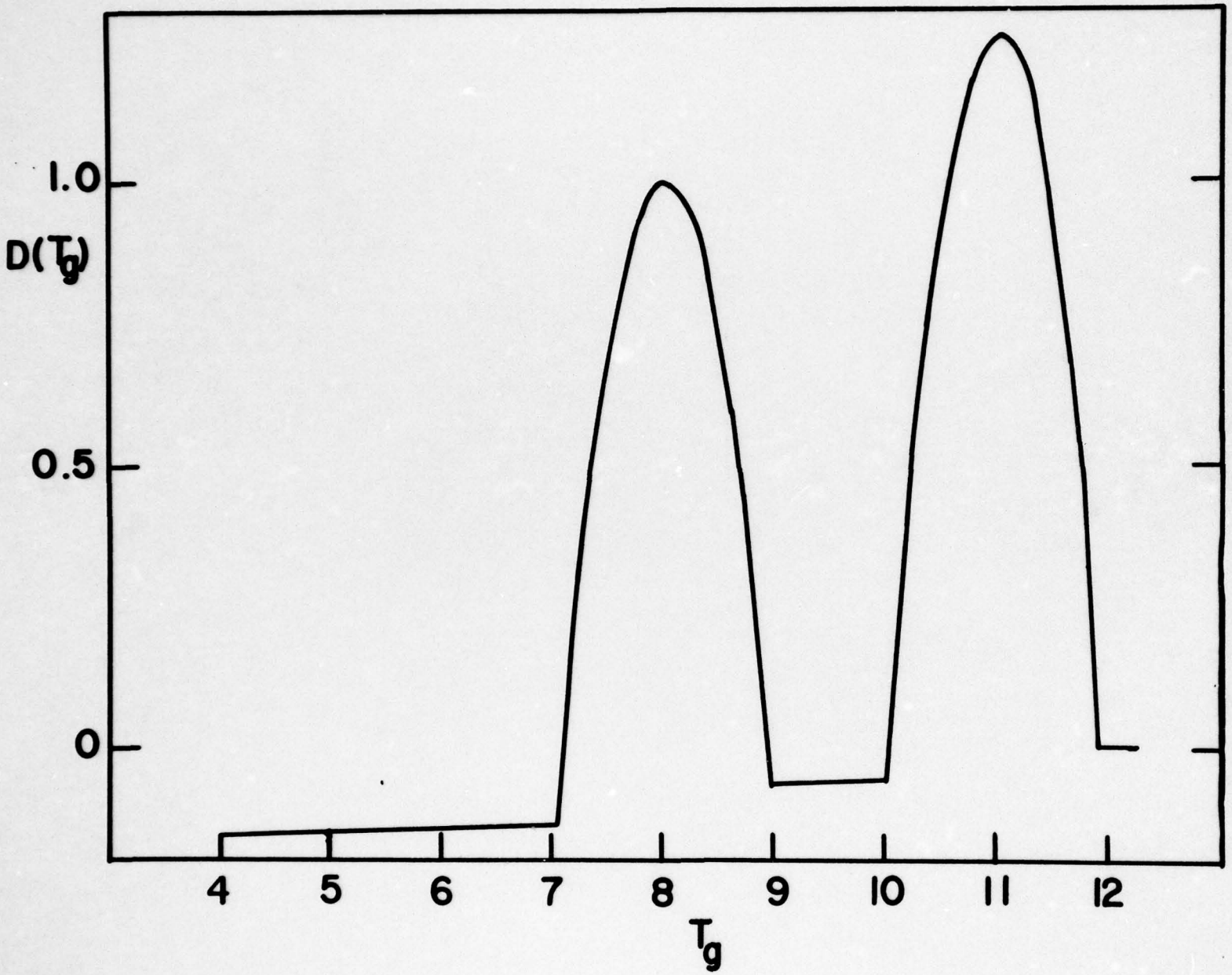

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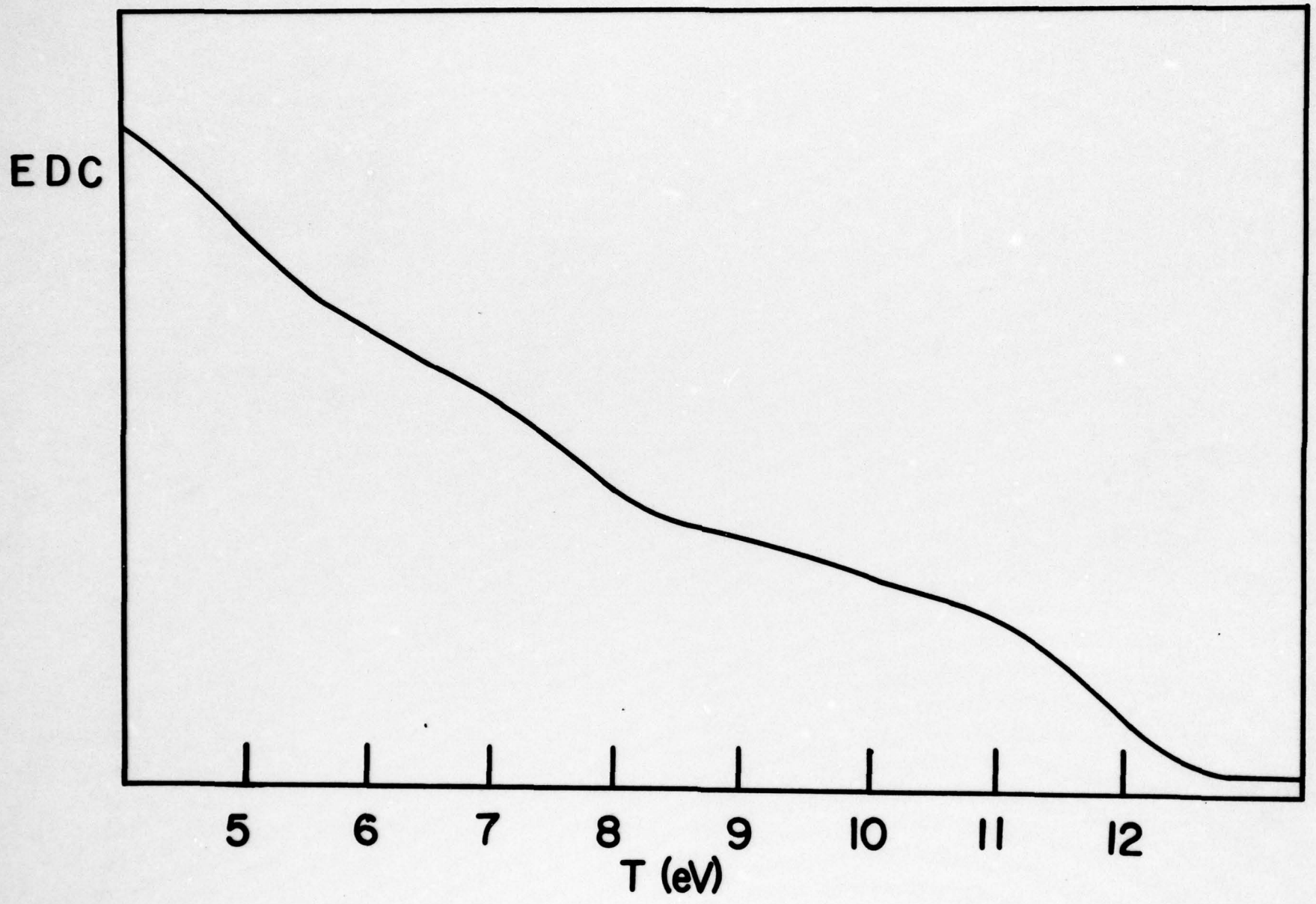

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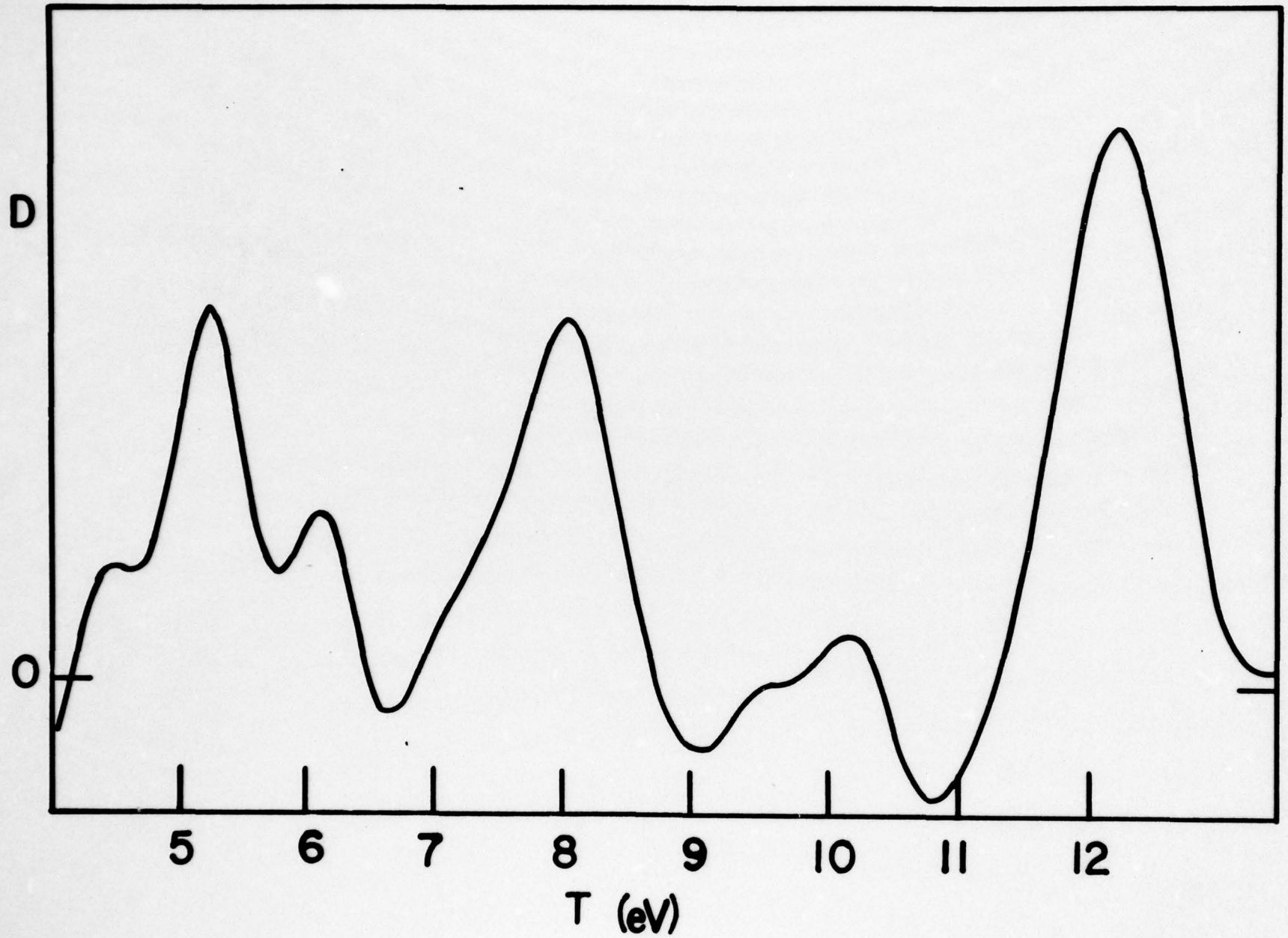

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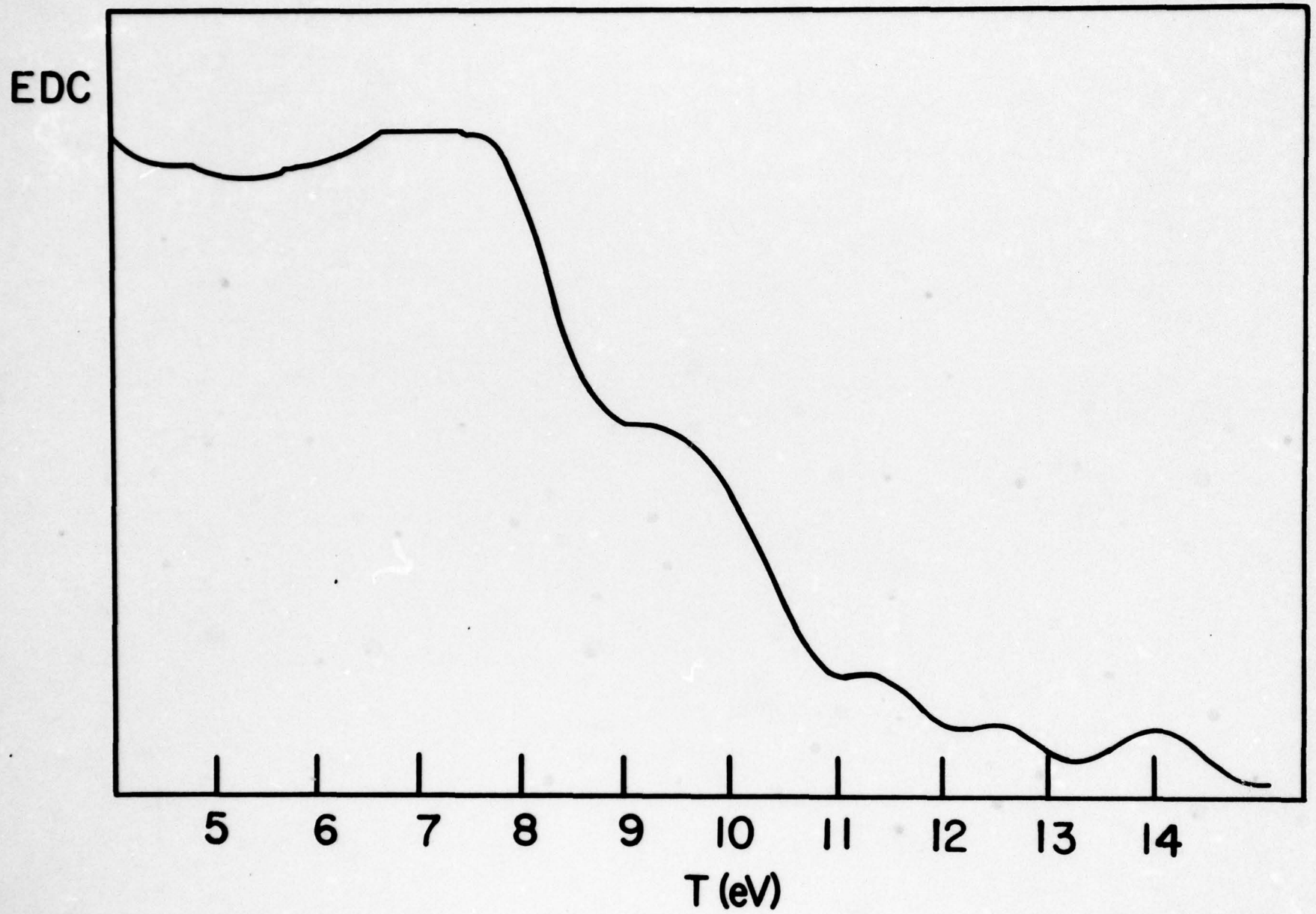
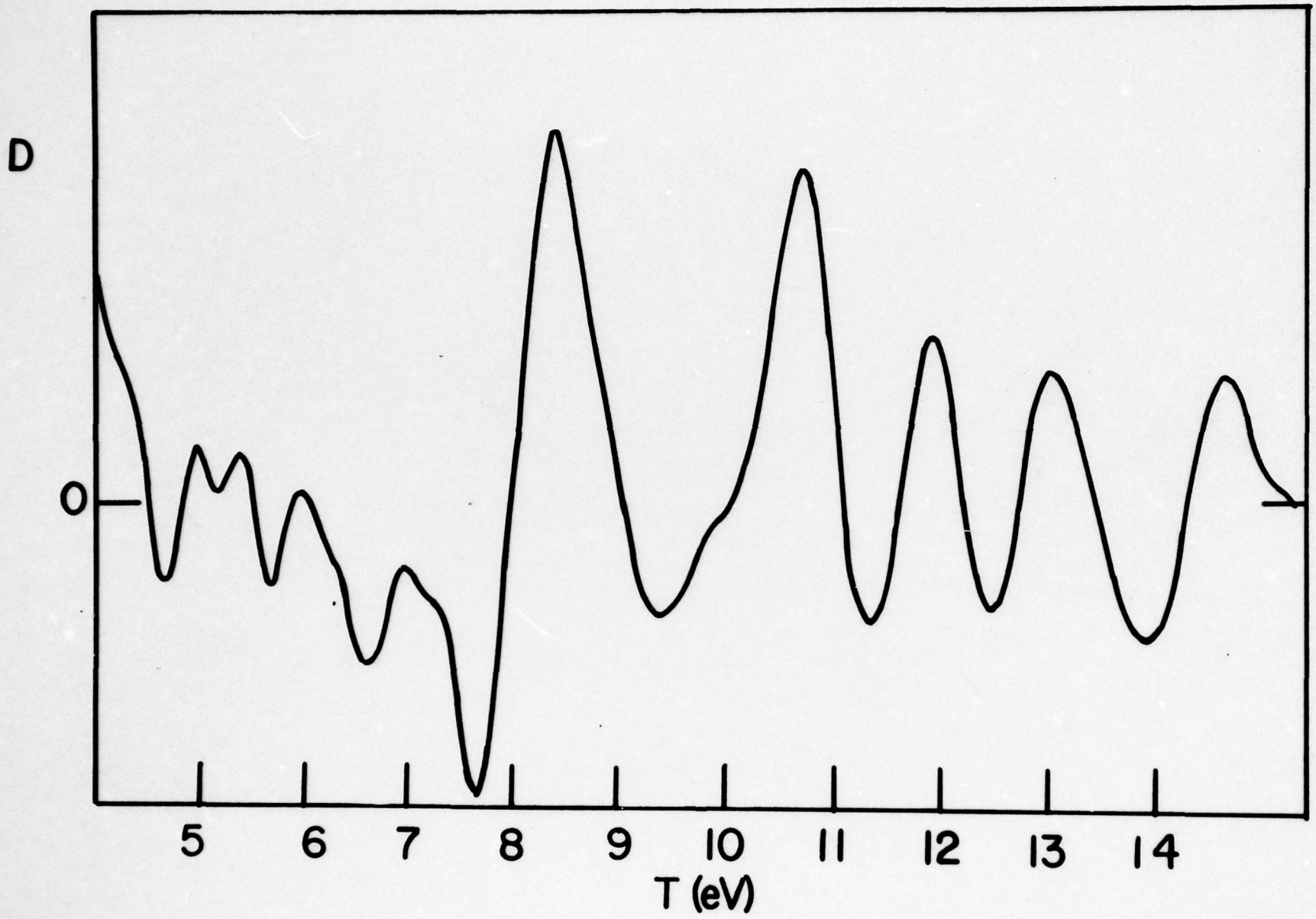


FIG. 7



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